

What we claim as our invention is:

1. A process of producing highly purified acetonitrile comprising (1) distilling crude acetonitrile in a first distillation column affixed with a first overhead  
5 reflux loop at a first pressure of at least 1 atmosphere to remove HCN, producing a first acetonitrile/water azeotrope and a first bottom product containing water, (2) distilling the first azeotrope in a second distillation column affixed with a second overhead reflux loop at a second pressure less  
10 than 1 atmosphere to separate the first azeotrope into a second bottoms product containing water and a second acetonitrile/water azeotrope having a greater acetonitrile concentration than the first azeotrope, (3) distilling the second acetonitrile/water azeotrope in a third distillation  
15 column affixed with a third overhead reflux loop at a third pressure above 1 atmosphere to produce a third acetonitrile/water azeotrope containing substantially all of the water from the second azeotrope, a third bottoms product comprising acetonitrile and heavy organics, and a side  
20 stream comprising highly pure acetonitrile, and (4) passing the highly pure acetonitrile side stream through an acidic ion exchange resin to further purify said highly pure acetonitrile producing highly purified acetonitrile wherein the reflux ratios in Steps 1, 2 and 3 are kept at greater  
25 than 3 to 1, greater than 3.4 to 1 and greater than 6.4 to 1, respectively.

2. The process of claim 1 wherein the reflux ratio in step 1 is between about 4.4:1.

3. The process of claim 2 wherein the reflux ratio in step 2 is between about 4.5:1.

5 4. The process of claim 3 wherein the reflux ratio in step 3 is maintained between about 8:1.

5. The process of claim 1 wherein the acidic ion exchange resin is selected to include a strong acid incorporating sulfuric acid functional groups.

10 6. The process of claim 1 wherein the acidic ion exchange resin is selected to include weak acid resin incorporating carboxylic acid functional groups.

7. The process of claim 1 further comprising distilling the purified acetonitrile recovered from the  
15 acidic ion exchange resin.

8. The process of claim 7 wherein the reflux ratio in step 1 is between about 4.4:1.

9. The process of claim 8 wherein the reflux ratio in step 2 is between about 4.5:1.

20 10. The process of claim 9 wherein the reflux ratio in step 3 is maintained between about 8:1.

11. The process of claim 7 wherein the acidic ion exchange resin is selected to include a strong acid incorporating sulfuric acid functional groups.

12. The process of claim 7 wherein the acidic ion exchange resin is selected to include weak acid resin incorporating carboxylic acid functional groups.